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ENVIRONMENTAL TRACE MATERIALS:
COMPUTER COUPLED RADIOACTIVATION ANALYSIS

Milton H. Feldman, et al

Environmental Research Laboratory
Corvallis, Oregon

December 1975

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ABSTRACT

A neutron activation laboratory with computer coupled equipment and procedures was established. The power of the methodology for Environmental Trace Material Analysis was demonstrated by analyzing various materials and included quality control interlaboratory comparisons. Samples ranged from sewage treatment plant sludges and marine sediments to fresh waters containing very low concentrations of molybdenum, and ores and fertilizers containing cadmium.

This report is submitted by the Corvallis Environmental Research Laboratory, Corvallis, Oregon, under the sponsorship of the Environmental Protection Agency.

SECTION I CONCLUSIONS

FEASIBILITY OF RADIOACTIVATION ANALYSIS

Radiation methodologies for environmental trace materials research, in particular neutron activation or the equivalent x-ray spectroscopic methods, are feasible at laboratories with access to suitable irradiation sources. Neutron activation analysis (NAA) is desirable for environmental tracer experiments because it is sensitive and uses constituent tracer nuclides rather than introducing other tracers.

EQUIPMENT

Equipment designated as shelf items and fairly unsophisticated computer programs are adequate for a good automated program of trace material environmental research and monitoring; however, the cost may be significant. The total cost of simple equipment described in this report was about \$40,000 (~1972), plus access to a time sharing computer system and a nuclear reactor. This estimate does not include the time and cost to prepare and evaluate efficiency of computer programs.

COMPARISON AND REPLICATIONS

The comparison of analytical results indicates that either atomic absorption (AA) or NAA can be used to analyze environmental trace materials. However, the minimum size of samples that can be analyzed differs greatly for the two methods. The use of higher flux (available at numerous reactors) enables treatment of much smaller samples than were handled in the development and routine work. Whereas, 100 mg samples were adequate with $\sim 10^{12}$, smaller samples can be assayed with flux $\sim 10^{14}$. In an investigation of molybdenum in lake waters, 10 ml

water samples irradiated at a flux of $\sim 10^{14}$ were shown to have molybdenum content ranging from ~ 0.68 to 0.81×10^{-9} gram/ml; typical AA procedures can only detect 2.0×10^{-5} gram/ml using a 200 ml sample and resorting to sample concentration techniques. Replication indicates adequate precision to study changes, if they occur, on the order of 25 percent at the microgram per gram level in 0.1 gram samples or, using higher flux reactors, in 0.001 gram samples.

ENVIRONMENTAL SAMPLE ASSAY

Automated environmental monitoring with graphic display of "finger prints" also may be carried out by NAA. For the sludge and sediment samples considered in this work, gold, silver, and chromium are useful elements for sludge tracing.

REFEREE METHODOLOGY

In addition to its general usefulness as a sensitive method for trace analysis, NAA frequently is used as a referee or comparison method. For example, phosphate ores and fertilizers were analyzed for heavy metal content. The Environmental Protection Agency (EPA) laboratory in Region X ascertained that some fertilizer and ores contained significant cadmium and requested NAA for comparison. Results were obtained from radioactivation and compared with the results from AA using wet chemistry procedures. As predicted by the observations of Bowen and Gibbons (1963), the results were typically parallel, but NAA reported lower values.

SECTION II INTRODUCTION

STANDARD PROCEDURES FOR ENVIRONMENTAL TRACE MATERIALS

Although various chemical analyses of environmental samples have acquired "standard procedure" status (Standard Methods, 1971) (Strickland, 1968) (ASTM, 1972), the requirements for analytical chemical information grow more stringent. Analysis of trace materials requires sensitivity and precision adequate not only to discern the trace materials of interest in surveillance sense, but also to enable following small changes in their chemical, physical, and biological fates and mechanisms. Such behavioral observations permit the computation (Morel, 1972) (Branica, 1968) and evaluation (Feldman, 1970) (Bowen, 1966) of the potential ecological influence of trace materials.

The procedures utilized must be applicable to the specific matrix and be amenable to some degree of routinization or automation since environmental problems frequently involve numerous samples to define a field, or to follow the behavior, and effect of a pollutant in the environment.

INTERCOMPARABILITY

Intercomparability of results of analyses from laboratory to laboratory and studies of so called "standard samples" have not been utilized where only environmental trends or changes have been studied; but such inter-comparisons and standard sample analyses are now considered essential (Feldman, 1964). (Bowen, 1964). It has been pointed out (Bowen, 1963) that historically the trace material analyst has tended to report lower values for trace elements as chemical sophistication advanced and that neutron activation analysis gives about as

uncontaminated and direct a result as is presently available. It should be recognized, too, that radioactivation procedures that utilize nuclide characterization give not only a readout, but also positive identification.

This is not only always the case where only a readout occurs as in atomic absorption and atomic fluorescence spectroscopy. Problems and interferences, such as the severe iron interference in zinc estimations (Kelly, 1973), or the sodium salt matrix interference with marine sample trace element determinations by typical atomic absorption procedures, require large blank corrections or prior chemical processing. But such chemical processing is not feasible in the case of trace materials or may lead to large, uncertain corrections dependent on numerous parameters that require elaborate empirical handling (Brooks, 1967).

RADIOACTIVATION, A GENERAL METHOD

Radioactivation analysis provides a multielement, nondestructive capability which meets the above provisos, for a fairly large group of elements (Bowen and Gibbons, 1963). It does require elaborate experimental facilities and is generally slower in output than atomic absorption. On the whole, its susceptibility to automation and computer coupling is better than most other procedures. Radioactivation does not measure speciation since it measures the whole element independent of chemical or physical form; it can distinguish between low concentrations whose differences may result from speciation experiments (Strohal, 1973).

SECTION III

ENVIRONMENTAL TRACER EXPERIMENT

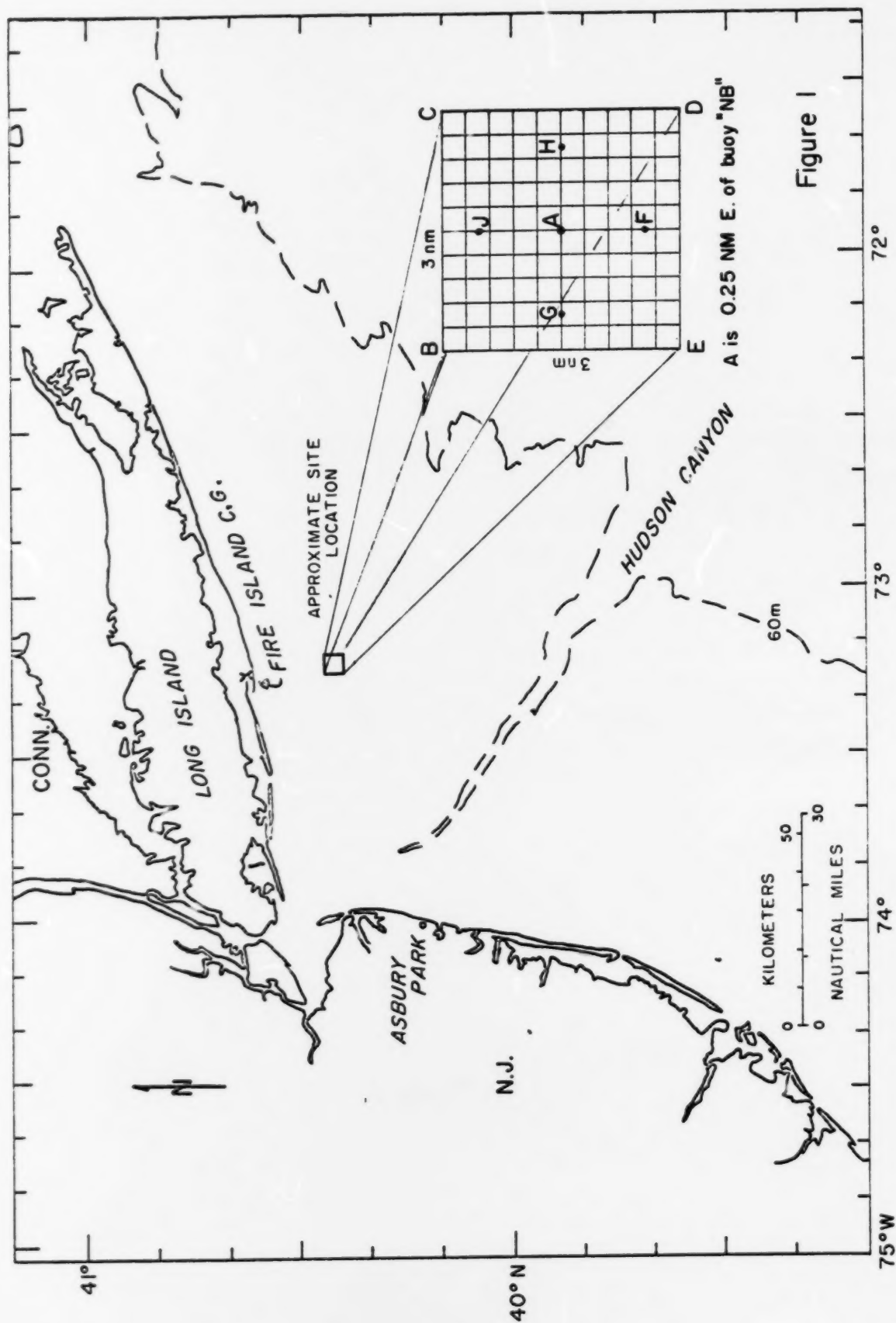
APPLICATION OF RADIOACTIVATION

Radioactivation--- specifically instrumental neutron activation analysis* (INAA) based on Ge(Li) crystal detection with pulse height analysis and computer coupled data handling and analysis---was used to estimate some trace elements in sludges from sewage treatment plants in the New York City area and in sediments from the New York Bight (Figure 1).

The principal objective of this work was to establish a trace materials analytical laboratory capability for environmental research with procedures based on computer coupled radioactivation, utilizing automated sample handling and computer data analysis. As part of the procedural development, analytical results were compared from the NAA laboratory at Corvallis, a central laboratory service group (LSB) at Corvallis, and the New York group (NYSTP) which operates the plants from which the sludge samples were obtained.

A second objective of this work was to identify one or more suitable constituent tracer nuclides in sludge that are not present at interfering levels in the sediments. Then, after placing experimental quantities of sludge on the ocean floor, it is feasible to follow the detailed movement and chemistry, of the sludge materials when studied over a long period of time.

* I am greatly indebted to the Oregon State Board of Higher Education, the Oregon State University, and Professor Chih Wang, Director of the OSU Radiation Center, for permission to use the Radiation Laboratory facilities with Program Director Status. MHF.



SECTION IV

THE EXPERIMENTAL PROCEDURE

SAMPLE SIZE

Samples typically were about 100 mg for convenient handling, although fractional mg samples can readily be analyzed at a slower pace and higher cost.

WEIGHING

The introduction of automated weighing with electronic readout simplified the laborious sample handling. Samples were placed in preweighed, numbered vials of polyethylene, quartz, or aluminum (Battistone and Feldman, 1966), then dried at 27°C. The weighings, on a Cahn Electrobalance, were electronically read out and recorded in a notebook or later were read out to a teletypewriter as well as indexed.

If circumstances require a preconcentration step (Joyner, 1967), (Feldman, 1967) (Rottschafer, 1971), or if the sample holder contributes trace materials in instrumental neutron activation analysis (INAA), a small blank may occasionally be necessary. It is generally the case that the entire sample containing the component sought is encapsulated, irradiated, and analyzed afterwards when interferences are minimized because the element sought has been made distinguishable from the natural elements present in the laboratory. The chief sources of contamination are from the equipment used to procure the environmental sample and especially from the laboratory dust itself.

IRRADIATION

In order to identify potentially useful constituent trace nuclides, four time groups of nuclides, irradiation and subsequent decay, and counting were considered. The utility of this sequence has been demonstrated (Dams, 1970) for air pollution studies. See Table 1 and Table 2. (Hughes 1958, et seq).

TABLE 1. IRRADIATION, DECAY, AND COUNTING TIME SEQUENCES

	t_{act}	t_{decay}	t^*_{count}
A	300 sec.	300 sec.	200-400 sec.
	300 sec.	900 sec.	1000 sec.
B.	8-16 hr.	20-30 hr.	2000-4000 sec.
	8-16 hr.	20 d.	4000 sec.

*This period is easily adjusted after a preliminary examination of the first sample of a set. The size of the adjustment, depends on the number in the set and the half life group.

TABLE 2. NUCLIDE SERIES CONSIDERED

0.1 ¹	1.0 ¹	10 ¹	100 ¹	t _{1/2} ²	α ³	ω ⁴	γ ⁵	AI ⁵	γ ⁶	AI ⁶	γ ⁷	AI ⁷	γ ⁸
Co 60m				10.5m	18	100	58.5	2.4	1332.5	0.025			
Se 79m				3.9m	0.36	23.5	95.9	12.2					
Ag 108				2.4m	35	51	619.0	1.8	433.8	0.54			
	Mn 56			2.6hr	13	100	846.7	100	1811.2	29.4	2112.6	16	
	Ni 65			2.56hr	1.5	1	1481.7	24.7	1115.4	14.8	366.5	4.93	
	Se 81m			57m	0.1	49.8	103.1	98.8					
				12.8hr	4.4	69	1345.8	0.48					38
	Cu 64			14hr	0.09	18.6	439.1	100					
	Zn 69m			26.4hr	4.5	100	559.1	44.6	657.1	6.24	1216.3	3.4	
	As 76			66.7	0.51	23	739.7	13.7	181.1	9.5			
	Mo 99			6.7hr	1	1.2	93.0	4.8					
	Cd 107			53hr	1.1	28.9	527.9	27.5	492.3	8.1			
	Cd 115			65hr	880	0.15	77.6	20	191.4	8.9			
	Hg 197			24hr	25	0.15	133.9	34.2					
	Hg 197m			27d	16	4.4	320.1	9.8					
	Cr 51			310d	0.8(14)	100	834.8	100					
	Mn 54			45d	1.1	0.31	1099.3	56.5	1291.5	43.5			
	Fe 59			5yr	19 + 18	100	1173.2	100	1332.5	100			
	Co 60			243d	0.5	49	1115.5	52.4					
	Zn 65			120d	30	0.87	264.7	59.5	136.0	57.2	279.5	24.6	
	Se 75			255d	3	49	657.7	94.4	884.7	75.1	937.5	34	
	Ag 110m			43d	0.14 + 1.1	29	934.1	2.5	1289.9	1.03			
	Cd 115m			60d	3.3	42.8	602.7	98					
	Sb 124			2.7d	100	100	411.8	96.8					
	Au 198			47d	4.0	30	279.2	81					
	Hg 203												35

1 - Group designated by half life region (hours)

2 - Precise half life

3 - Cross section, barns

4 - Abundance

5 - Principal gamma

6 - Second most prominent gamma

7 - Third gamma

8 - Annihilation gamma as % of decays

COUNTING

After suitable irradiation and decay a set of samples is placed in the counting and recording system consisting of a Nuclear Chicago, Model 1035, mechanical sample changer, which is coupled in and controlled by a Nuclear Data 2200 Pulse Height Analyzer (PHA). See Figure 2. The PHA records signals (pulses from the 15% efficiency, 2.3% resolution, Ge(Li) crystal*) in its 4096 channel memory through a 50 megahertz analog digital converter (ADC). At the conclusion of a preset count period the data are read out into either:

- a. A telephone line feeding directly to the computer⁺.
- or b. A magnetic tape bulk data storage system based on Kennedy tape transport model 3112.

Routine analysis utilizes the tape; experimental development work utilizes either the wire or the tape or both. The tape is hand carried to the computer at the end of a sequence of samples for subsequent interrogation and printout of tables of data as allowed for in our computer programs.

COMPUTER COUPLED NAA

The computer has proved very practical and helpful to handle large quantities of data generated by the automated weighing, counting,

* Compared to a 3" NaI crystal and using a Co⁶⁰ 1.33 MeV line; by the manufacturer, EDAX International, Prairie View, IL.

⁺ Oregon State University (OSU), Control Data Corporation 3300 + OS3 operating system.

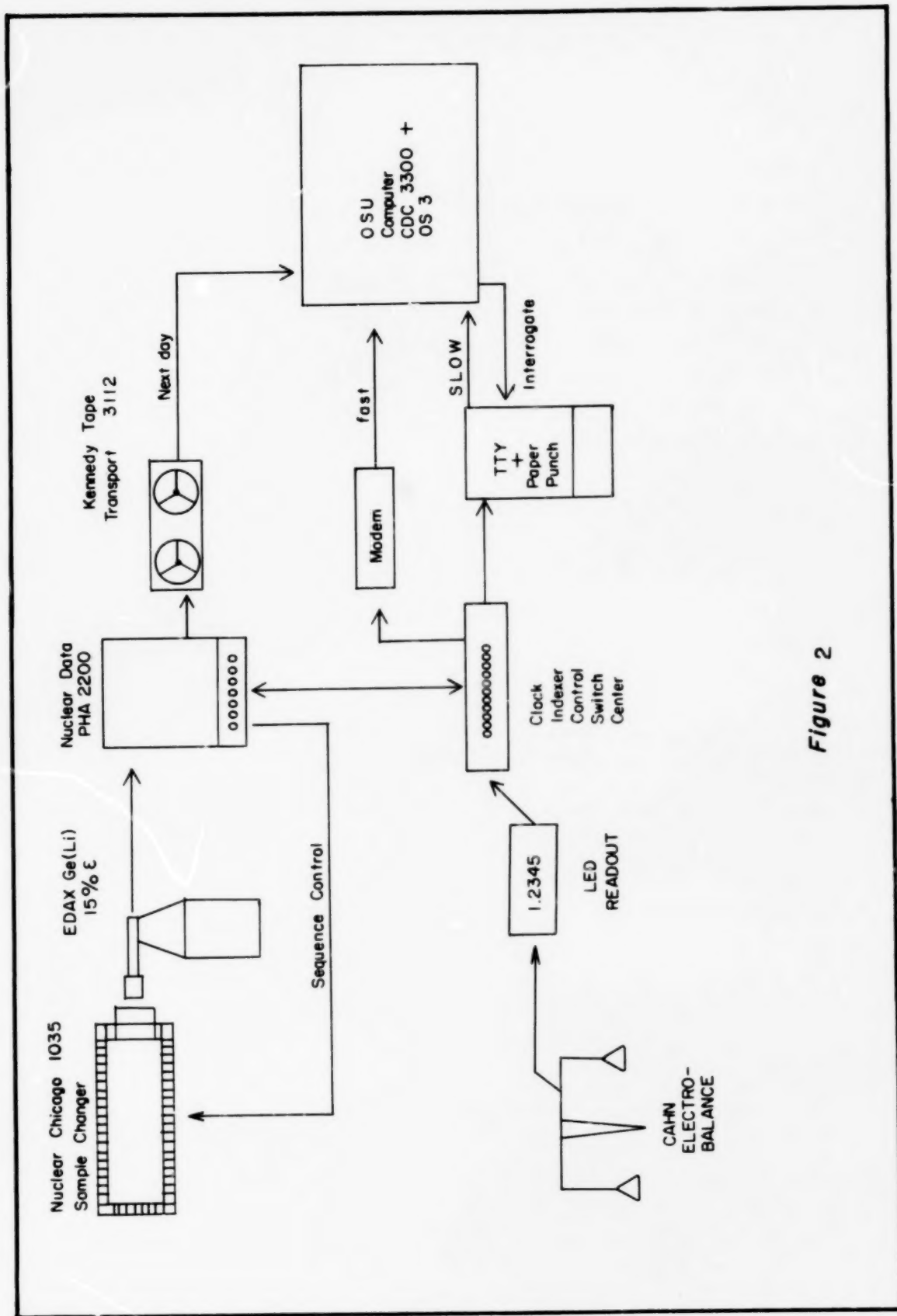


Figure 2

timing, accumulation and transmission of data from 4096 channels of the PHA memory. Originally suggested as a desirable development a number of years ago, (Fite, 1961), its development only became practical for analytical chemistry with the advent of large Ge(Li) crystals. It should be recognized that instrumental analysis alone still cannot offer all the criteria sometimes required by the analytical chemist, but this is not to gainsay its great convenience. Many descriptions of detailed and very ambitious programs* for computer coupled neutron activation analysis have been published (Gunnik, 1972) for specialized usages where sufficient numbers of chemical plant samples and work load justify the specificity of program.

Our relatively unsophisticated programs which allow diversity of application were each generated, as the need became obvious, as a convenience to allow a technician to carry out simple laboratory procedures and to have voluminous data handled for him as a substitute for rather pedestrian data printing and hand computation. The program items presently available allow for:

1. Area calculation of any "regions of interest" whose boundary channel numbers are specified to the computer. These areas are corrected for background, compared to standards similarly specified, and subjected to standard deviation statistical examination. This program, particularly useful for tracer experiments that generate many samples containing a few known radionuclides, is also useful for computer interrogation that experimentally evaluates a specific nuclide procedure for development.

*Some such large detailed programs, carried out essentially in real time and allowing for numerous samples and contingencies, effectively tie up large computers. Unfortunately, even with fast front end and high speed ADC, the data uptake rate and PHA is still a bottleneck.

2. Calibration of the 4096 channels of the PHA for a range of energies. This uses a set of standard materials (Dams, 1968) (Filby, 1970). (Gunnik, 1969) or, more often, an element like europium-152 with several well known gammas over the range of energies generally used. Comparison of any unknown peak is by interpolation between the closest peaks used in the calibration.

3. Peak searching by observation of changes of slope between successive sets of channels by the procedure of Borchardt (1970), locates the edges of the peak, the peak energy, estimates the area of the peak, corrects for background, and estimates the standard deviation of the area. Printout of this information comprises the essential data of our system for quantification. See Table 3. Program item 4 which involves a degree of judgment is of equal importance in developing reliable procedures.

4. The computer program has access to a very complete library of data on thermal neutron generated activities.* A printout of relevant data on both sides of the energy of the peak of interest over any desired energy span is easily evoked and printed out. Along with the gamma energies there is printed out: nuclides, half lives, abundances, cross sections, accompanying gammas. See Table 4. This information allows, in most instances, decision as to identity, as well as quantitation; in some instances additional information from decay is required. Quittner (1972) points out that energy and decay data generally are adequate for positive identification as well as quantitation.

* We are greatly indebted to R. H. Filby and his colleagues at Washington State University for making available to us this valuable compilation of data.

TABLE 3. PEAK SEARCH PRINTOUT
Sediment A96M1B

UNKNOWN: J5T29

07/06/73 12:41 PM

No.	PCL ^a	Energy(keV)	Area	Error ^b	Wgt ^c	Error	
1	33	19.7	15855	538	0	0	
2	169	87.3	1719	326	0	0	
3	178	91.8	1109	253	0	0	
4	185	95.3	1955	263	0	0	
5	192	98.7	4094	287	0	0	
6	239	122.1	6355	471	0	0	
7	262	133.5	7903	285	0	0	
8	268	136.5	2298	239	1.11	.12	
9	287	145.9	8186	341	0	0	
10	315	159.9	548	171	0	0	
11	351	177.7	1338	356	0	-	
12	381	192.6	3156	347	0	0	
13	392	198.1	2056	293	0	0	
14	413	208.5	1457	332	0	0	
15	429	216.5	1409	265	0	0	
16	486	244.8	729	163	0	0	
17	526	264.7	1923	285	1.45	.22	
18	556	279.7	1080	264	0	0	
19	621	312.1	7052	256	0	0	
20	637	320.1	4709	301	5.20	.34	
21	678	340.5	726	211	0	0	
22	685	344.0	4233	221	0	0	
23	502	352.5	1092	226	0	0	
24	822	412.3	7513	229	.31	.01	
25	963	482.0	6411	224	0	0	
26	991	495.8	1263	178	0	0	
27	1209	604.6	880	163	0	0	
28	1218	609.1	883	173	0	0	
29	1316	658.2	3023	170	2.65	.15	
30	1530	765.0	895	202	0	0	
31	1566	782.9	168	115	0	0	
32	1592	795.9	575	163	0	0	
33	1670	834.8	1857	207	0	0	
34	1771	885.2	1135	183	1.75	.28	
35	1779	889.2	15434	263	0	0	
36	2096	1048.0	69	44	0	0	
37	2199	1099.6	13521	188	0	0	2.76 ^d + .031
38	2242	1121.1	14530	248	0	0	
39	2346	1173.2	6456	137	2.28	.05	
40	2581	1291.3	9244	124	0	0	2.34 ^d + .031
41	2662	1332.0	5998	105	2.41	.04	
42	2688	1345.1	26	11	0	0	
43	2915	1459.1	1392	54	0	0	
44	3092	1548.1	22	8	0	0	
45	3166	1585.3	29	12	0	0	
46	3296	1650.6	20	7	0	0	
47	3440	1723.0	40	33	0	0	
48	3636	1821.5	21	9	0	0	
49	1055	527.4	34	47	7.92	11.18	

^a peak channel

^b error (standard deviation of the count)

^c weight of nuclide by comparison to a set of standards similarly counted

^d weight of nuclide by comparison to a separate standard by manual computation

TABLE 4. CANDIDATE NUCLIDE PRINTOUT

Energy	Nuclide	Half-Life	X-Section	ABUN	Association Gammas	
Peak Energy = 338.70 ^a						
335.8	51-SB-124	6.03E+01D	3.30E+00	42.75	603	1691
336.0	70-YB-169	3.18E+01D	1.10E+04	.14	64	197
336.2	49-IN-115M	4.50E+00H				
336.6	31-GA- 72	1.41E+01H	5.00E+00	39.80	834	2202
337.3	65-TB-160	7.21E+01D	4.60E+01	99.99	879	299
338.3	32-GE- 77	1.13E+01H	1.00E+01	7.67	264	211
339.9	61-PM-151	2.78E+01H			168	275
340.0	67-HO-166M	1.20E+03Y	1.00E+00	99.99	185	811
340.5	91-PA-233	2.70E+01D			312	300
341.6	71-LU-177M	1.61E+02D	1.00E+00	2.60	208	229
Peak Energy = 846.20 ^a						
843.8	12-MG- 27	9.46E+00M	2.70E-02	11.29	1015	171
844.3	32-GE- 77	1.13E+01H	1.00E-01	7.67	264	211
844.7	46-PD-111M	5.50E+00H	4.00E-02	11.81	172	70
844.9	52-TE-129M	3.41E+01D	1.70E-02	31.79	696	730
845.4	63-EU-154	1.60E+01Y	3.20E+02	52.23	123	1274
845.8	36-KR- 87	7.60E+01M	6.00E-02	17.37	403	2556
846.5	54-XE-125	1.68E+01H	1.10E+02	.10	188	243
846.7	25-MN- 56	1.55E+02M	1.33E+01	99.99	1811	2112
848.9	61-PM-151	2.78E+01H			340	168

^a Where the observed peak energy 338.70 was entered, the computer printed the data on known species in that vicinity i.e., energy, nuclide, half life, cross section, abundance of isotope and associated gammas.

Decay information of a given peak or set of peaks in a sequence of counts is always desirable in developing procedures for samples of a newly given set. The use of the tape for recording a set of samples' changing spectra with time by repetitive counting in the mechanical sample changer gives a set of data that can easily be used for this purpose. Thus, suppose 18 samples are counted ten times in a 48 hour period. Then the tape data in the computer are queried for a given peak (region of interest per program item 1) in repetitive spectra for a given sample or samples and the results plotted using a simple least squares statistical evaluation to obtain the decay constant for each nuclide peak of interest.

See Figure 3a, 3b. The intercept, at time 0 is used for quantitative comparisons, while the positive identification is obtained from the combination of the $t_{1/2}$ of program item 5, with the E_{γ} obtained in program item 3 and the candidate nuclide information printout of program item 4.

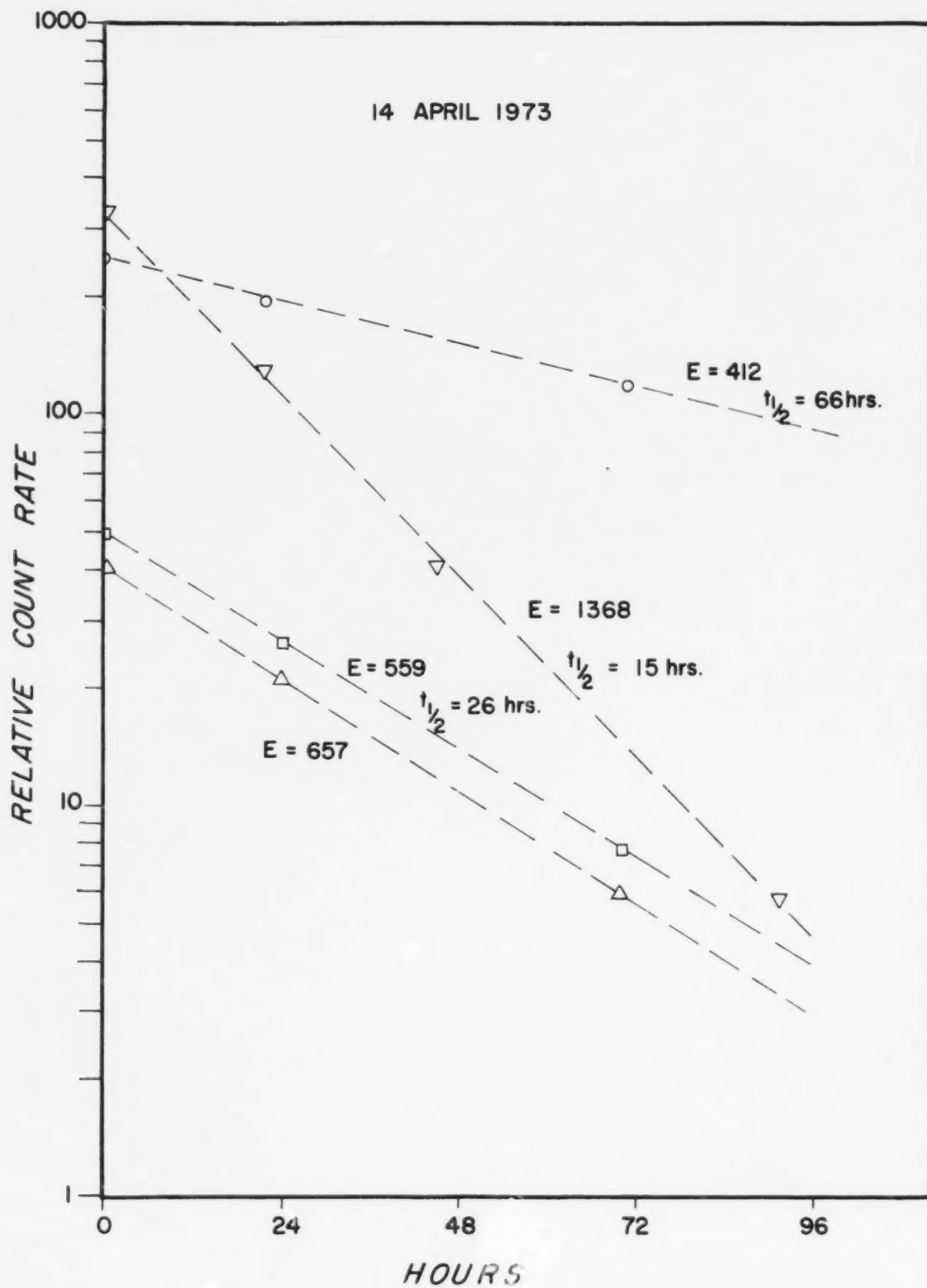
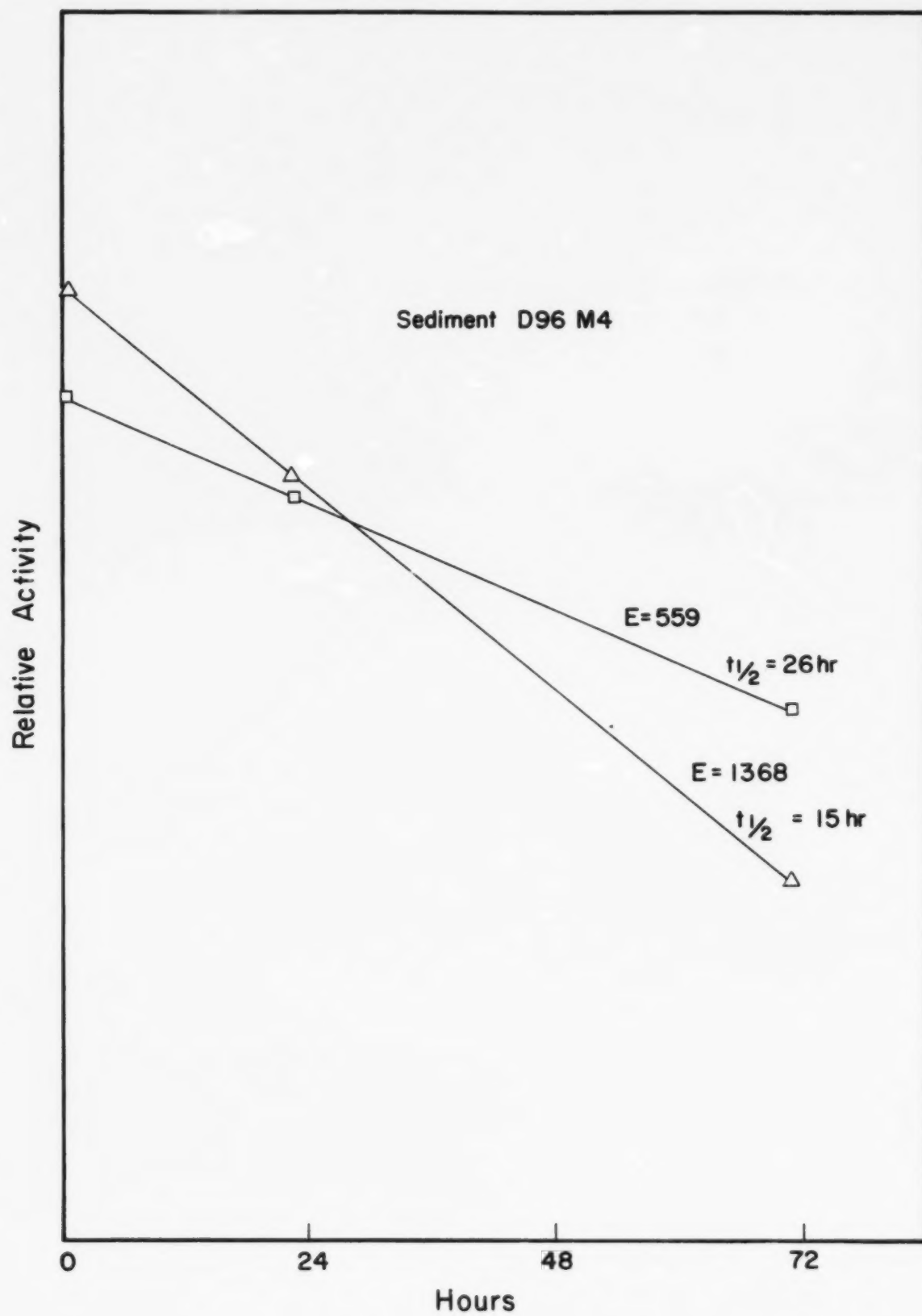


Figure 3a



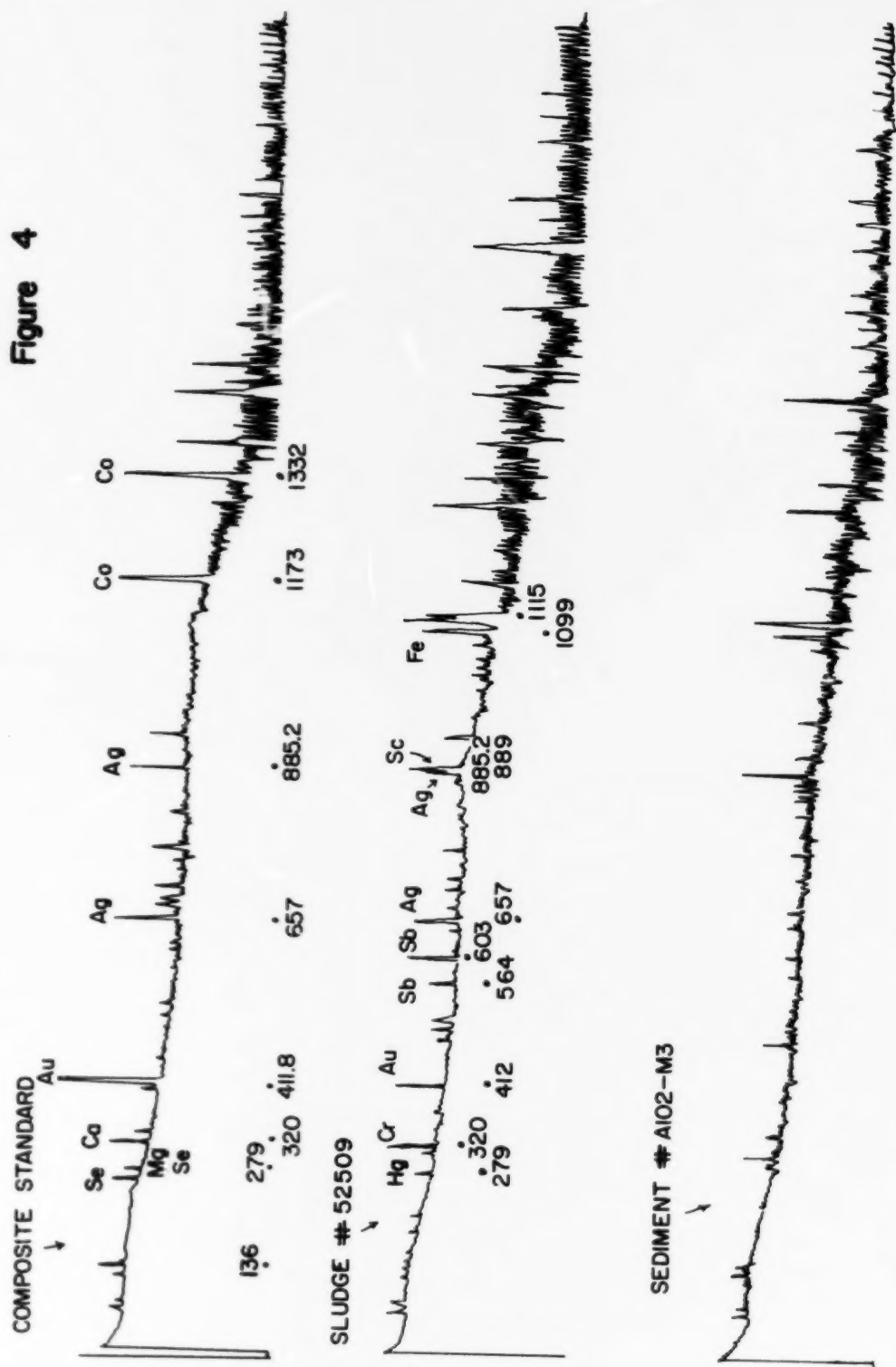
SECTION V

ENVIRONMENTAL MONITORING BY RADIOACTIVATION METHODOLOGIES

"Finger printing" and detection of very small changes in environmental samples is an important spinoff of neutron activation procedures easily presented by analog (graphic) display of the data. While not quantitative, as are the procedures described above, it does allow quick display of spectroscopic "finger prints." Generally such a display offers more information than can be readily grasped by eye and understood. However, the analog display considered as a picture can be very useful in a monitoring sense since changes or differences can be noted and later determined precisely by recall from the tape storage system and computer interrogation.

For example, planning sessions to evaluate a proposed experimental site raised the question: Since the size of sediment particles changes from point A to point B, accompanied by apparent changes in benthos are the two changes related or is this caused by a chemical change? Graphic display (see typical sediment spectra in Figures 4 and 5) allowed prompt application of chemical information to this question. The two actual spectra showed no obvious chemical difference, so biological differences seemed more likely to be related to particle size differences.

Figure 4



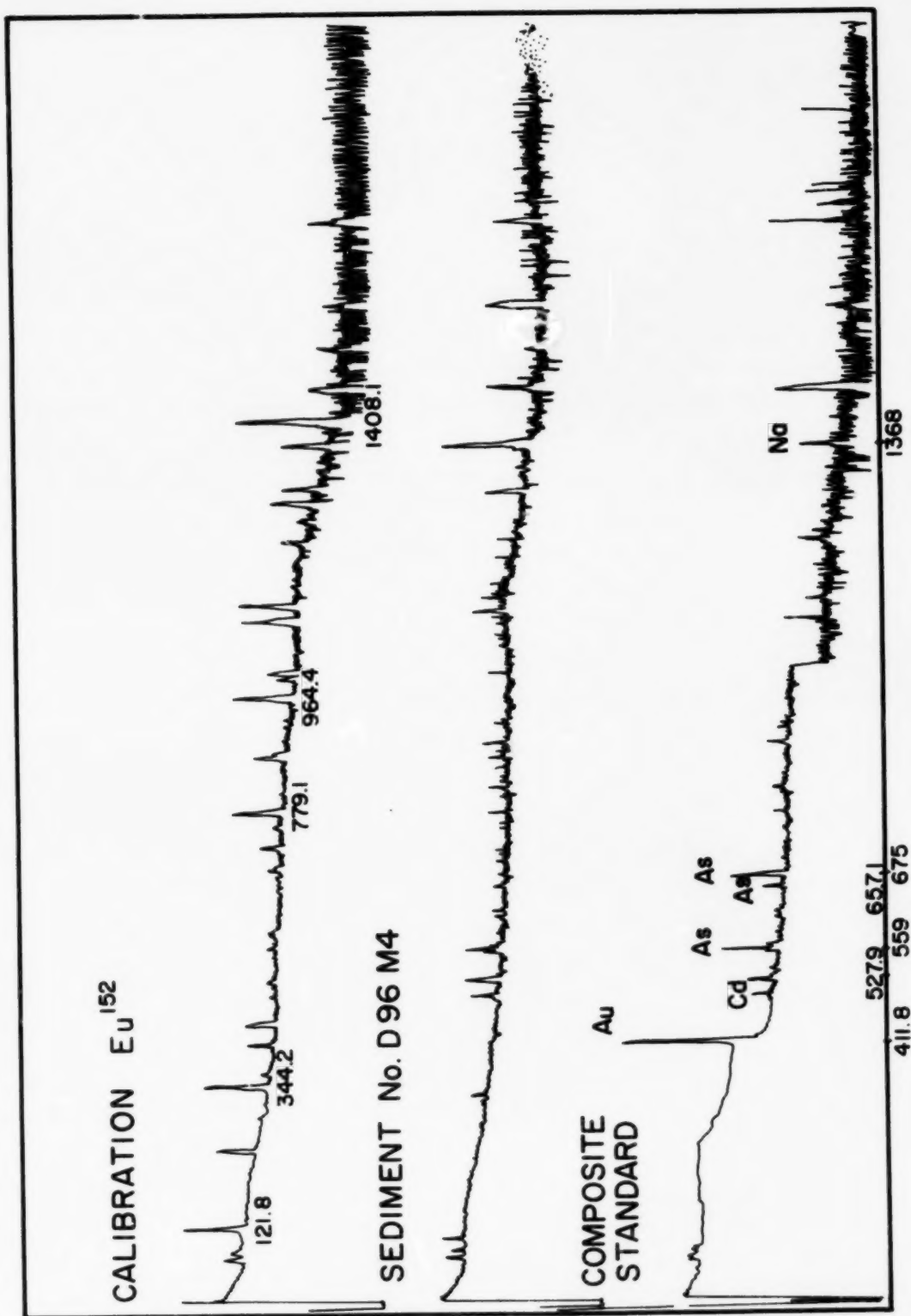


Figure 5

SECTION VI EXPERIMENTAL RESULTS

SAMPLES

Samples for analysis included sewage treatment plant sludges from various New York City area plants* and sections of 4" cores⁺ from the N. B. Buoy area (lat. 40°24'N, lon. 73°11'W) about 15 miles south of the Fire Inlet on the south shore of Long Island. See Figure 1.

In Corvallis the Laboratory Services Branch (LSB) used AA and the NAA laboratory used INAA to analyze the sludge samples. A parallel set of monthly composite sludge samples, by the New York area sewage treatment plant operators (NYSTP) used AA to analyse.

Thus, while the sampling, storage, transport, and analytical approach and actual nuclides measured are not identical for all three analytical groups there is enough overlap to make valid comparisons.

The sampling procedures of all three groups are considered reliable and, in view of the substrate's variable nature, quite adequate for these analytical purposes. Because LSB and NYSTP analyzed "wet samples" and dried a parallel sample to get dry weight equivalence, their numerical results have been made comparable with information from the total solids determinations.

* Obtained through the cooperation of Richard Dewling, Al Bromberg, and Dr. Richard Spear of EPA, Region II.

⁺ Obtained for us by various members of our staff, Dr. R. Swartz, A. Teeter, W. DeBen.

RANGES OF VALUES NAA/LSB

Table 5 compares some early NAA results with LSB results from well practiced AA procedures to show the range of values from eight sludge samples analyzed for various nuclides. No comparison is made when either LSB or NAA did not assay that nuclide. Evidently the two methods produce reasonably comparable results for aliquots taken from the same samples.

DETAILED ANALYSES---NAA, LSB, NYSTP

Table 6 compares a larger set of detailed data from NAA, LSB, AND NYSTP analyses. Since NAA and LSB used closely related samples, their results compared more closely than the NYSTP results. The NYSTP group analyzed a monthly composite; consequently its single result is tabulated against several NAA and LSB results from several samples variously collected during that given month.

SEDIMENTS

Table 7 shows sediment results analyzed by NAA alone. Remembering that the lower intensity peaks could be analyzed, the primary purpose here is to establish that gold, chromium and silver, if present, are low, and may be used as tracers within the sludge. Therefore, a--- or (blank) only means that the nuclide activity is low or has not been estimated. Table 8 shows the results from four sections of a core, each section divided into three equivalent samples and subjected to increasing amounts of washing and, incidentally, to increased amounts of handling. Replication shown in this table indicates that changes on the order of 25% at the microgram per gram level in 0.1 gm samples, or using high flux levels with 0.001 gm samples, could be studied.

TABLE 5. RANGE OF TRACE ELEMENT CONCENTRATIONS IN SLUDGE
(eight samples from N.Y. area)

	LSB	NAA
	(ppm dry weight)	
Cd	10-400	
Cr	200-800	400-1800
Co	7-20	15-36
Cu	800-1800	550-900
Fe	$\sim 10^4$	$[\sim 10^3]^a$
Pb	17-860	
Mn	240-560	200
Hg	10-20	$[21-37]^b$
Ag		6-13
Au		2-4
As		5-13 ^c
Se		0.5-1

^a Calc on Ag standard in absence of Fe Standard

^b Calc on Cr standard in absence of Hg standard

^c Average As was 9 ppm; extremes 5, 13 in eight sludges

Table 6a
SLUDGE INTERCOMPARISON

Sample	Location	Date	Se (136)	Hg* (279)	Cr (320)	Au (412)	Ag (658)	Co (1173)	Fe mg/g (1099)	Mn (847)
NAA LSB NYSTP	Tallman Island	6 July 72 July Ave.	0.54±.26	22.3 8.9 15.0	964± 71 780 910	2.18±.19	10.6±.2	22.7± .8 15.5	20.5 20.0	259±2.3 240
NAA LSB NYSTP	Tallman Island	11 July 72 (ave)	0.95±.35	26.9 9.4 15.0	1413±104 820 910	3.76 .44	13.5± .3	35.7±1.1 17.0	20.0 20	242±2.2 240
NAA LSB NYSTP	Jamaica Bay	11 July 72 (ave)	0.92±.25	28.9 11.0 72.0	586± 44 350 460	1.91±.24	8.48±.3	14.3±.67 14.0	22.0 19.0	350 ±2.0 560
NAA LSB NYSTP	Tallman Island	23 July 72 (ave)	0.66±.21	21.2 12.0 15.0	1050± 78 765 910	2.06±.26	10.5±.2	26.7±1.0 16.0	20.0 20.0	215 ±1.5 275
NAA LSB NYSTP	Jamaica Island	21 July 72 (ave)		9.2 72.0	300 460	2.85±.49		11.0	22.0	415 ±2.7 430
NAA LSB NYSTP	Tallman Island	28 July 72 (ave)	0.46±.18	28.9 8.3 15	428± 33 980 910	3.94±.28	5.51±.2	24.5± .95 16.0	20.0 20.0	570 ±3.1 240
NAA LSB NYSTP	Jamaica Island	28 July 72 (ave)	0.72±.20	36.7 5.9 72.0	562 ± 42 325 460	3.07±.18	6.90±.2	19.7± .72 11.0	20.0 19.0	258 ±1.8 430
NAA LSB NYSTP	Tallman Island	4 Aug. 72 (ave)	0.67±.25	26.1 12.0 15.0	1762±130 980 1270	4.34±.45	12.8±.3	31.9±1.1 7.0	18.0 26.0	189 ±1.6 225

*Calculated from standard Cr in absence of Hg standard
 **Numbers in parentheses under nuclide are approximate
 energy of gamma observed in kev
 All results in ppm dry weight basis except Fe

Table 6b
SLUDGE INTERCOMPARISON

Sample	Location	Date	Se (136)	Hg (279)	Cr (320)	Au (412)	Ag (658)	Co (1173)	Fe mg/g (1099)	Mn (847)
NAA LSB NYSTP 39501	Tallman Island	11 Aug. 72 (ave)	5.17±1.5	11.6±1.2 55.0	290 1272	7.29±.06	30.8±2.5	2.99±.09	26.0	449±5
NAA LSB NYSTP 39502	Jamaica Bay	11 Aug. 72	3.80±1.5	10.3±.85 55	704±22 550	4.62±.05	56.9±43	1.94±.08	28.0	278±4
NAA LSB NYSTP 39503	Tallman Island	25 Aug. 72	5.89±1.5	10.0±.94 55.0	714±23 1272	5.96±.05	58.7±4.4	2.27±.08	26.0	310±2
NAA LSB NYSTP 39504	Jamaica Bay	25 Aug. 72	5.51±1.7	10.7±1.10 46.0	325±11 550	4.53±.05	30.6±2.4	2.49±.10	28.0	473±3
NAA LSB NYSTP 39505	Tallman Island	1 Sept. 72	9.02±2.4	11.7±1.3 18 44	757±24 1200 818	5.79±.06	72.5±5.6	3.35±.12 7.5	17.0 11.0	288±2 280
NAA LSB NYSTP 39506	Jamaica Bay	1 Sept. 72	4.70±1.3	11.9±.97 10.0 116	363±12 476 1180	6.11±.04	32.2±2.5	2.38±.08 10.0	16.0 21.0	447±2 476
NAA LSB NYSTP 39507	Tallman Island	13 Sept. 72	5.04 1.8	11.1±1.4 11.0 44	755±24 1200 818	8.59±.07	57.4±53	3.22±.11 8.1	16.0 11.0	343±3 343
NAA LSB NYSTP 39508	Jamaica Island	13 Sept. 72	4.01±1.6	11.1±1.2 5.0 116	368±12 500 1180	10.1±.07	26.5±2.6	2.78±.09 6.5	15.0 21.0	464±3 395
NAA LSB NYSTP 39509	Tallman Island	14 Sept. 72	5.34±1.7	8.39±.81 7.9 44.0	755±25 1100 818	5.80±.05	56.3±5.2	2.67±.09 6.8	12.0 11.0	273±2 272
NAA LSB NYSTP 39510	Jamaica Bay	14 Sept. 72	2.92 1.3	10.3±.91 116	363±12 410 1180	4.21±.04	29.1±2.7	2.40±.08 11.0	14.0 21.0	

Table 6c
SLUDGE INTERCOMPARISON

Sample	Location	Date	Se (136)	Hg (280)	Cr (320)	Au (412)	Ag (658)	Co (1173)	Fe (mg/g) (1099)
NAA LSB NYSTP	Tallman Island	21 Sept. 72	3.08±1.2	8.75±.89 18.0 44.0	667±21 100.0 818	5.8±.07	49.1±5.1	254±.08 7.9	17.0 10.9
NAA LSB NYSTP	Jamaica Bay	21 Sept. 72	6.07±2.4	11.4±1.2 12.0 116.0	417±14 62 1180	3.81±.05	32.9±3.2	1.34±.15 12.0	16.0 21.0
NAA LSB NYSTP	Jamaica Bay	28 Sept. 72	1.34±.94	10.4±1.2 9.2 116.0	288±27 93 1485	4.17±.06	73.8±.10	2.45±.09 9.4	19.0 21.0
NAA LSB NYSTP	Tallman Island	6 Oct. 72	3.56±1.0	1.45±.61 21.0 57.0	612±27 93 1485	4.58±.05	58.1±.08	2.39±.07 10.0	15.0 28.0
NAA LSB NYSTP	Jamaica Bay	6 Oct. 72	3.31±1.1	7.48±.75 35.0 42.0	244±10 44 540	3.81±.07	96.6±.07	1.74±.07 9.1	15.0 26.0
NAA LSB NYSTP	Jamaica Bay	13 Oct. 72	3.90±1.3	8.59±.68 19.0 42.0	283±13 50 540	6.19±.05	99.6±.09	2.46±.08 7.7	16.0 26.0

Table 6d

SLUDGE INTERCOMPARISON

Sample	Location	Date	Se (136)	Hg (200)	Cr (320)	Au (412)	Ag (658)	Co (1173)	Fe (mg/g) (1099)
NAA LSB NYSTP	Rockaway Island	8 Nov. 72	4.49±2.2	31.0 275	202±15 285 222	1.79±.08	83.5±3.4	5.24±.46	18.1±.4 21 10
NAA LSB NYSTP	Coney Island	8 Nov. 72	2.89±1.9	20.4 35.0	346±22 327 242	2.33±.05	71.2±3.1	6.71±.44	18.0±.4 16.3 14.0
NAA LSB NYSTP	Coney	14 Nov. 72	4.44±1.9	17. 35	355±22 442 242	2.43±.04	74.1±4.0	6.74±.50	17.6±.4 17.7 14.0
NAA LSB NYSTP	Rockaway	24 Nov. 72	3.56±.10	9.3 27.5	107±7.1 112 222	0.93±.03	30.4±1.7	5.04±.32	17.3 17.0 10.0
NAA LSB NYSTP	Coney	24 Nov. 72	4.14±1.9	10.7 35.0	236±15 291 242	4.44±.07	53.5±2.4	5.59±.41	14.3±.3 14.2 14.0
NAA LSB NYSTP	Rockaway	29 Nov. 72		6.3 275	34±5.6 38 222	0.25±.03	10.6±1.4	2.27±.39	4.99±.3 6.0 10.0
NAA LSB NYSTP	Coney	29 Nov. 72	4.29±2.9	12.5 35	297±15 341 242	2.74±.09	66.6±4.7	7.54±.80	19.8±.5 19.1 14
NAA LSB NYSTP	Rockaway	6 Dec. 72		8.8 32	59.9±5.2 70 68	0.58±.06	22.8±1.9	0.50±.26	7.03±.3 8 12
NAA LSB NYSTP	Coney	6 Dec. 72	7.73±2.5	18.5 28.0	291±15 336 182	2.98±.07	68.3±3.9	6.35±.65	17.9±.5 18.7 14
NAA LSB NYSTP	Rockaway	13 Dec. 72		9.3 32	31.6±3.7 100 68	0.22±.05	10.9±1.5	3.91±.49	4.40±.3 4 12
NAA LSB NYSTP	Coney	13 Dec. 72	3.35±1.4	12.7 28	294±12 327 182		74.4±3.7	5.61±.41	17.8±.4 17.8 14

TABLE 7a

Sediment Core Section Analyses

Location	Core Cut	Date	Sc (136)	Sc (264)	Cr (320)	Au (412)	Ag (764)	Ag (937)	Co (1172)	Co (1332)	Fe (mg/gm) (1009)	Fe (mg/gm) (1291)	Sc (899)	Sc (1120)
E99	M1	2/21/73	8±.5	8±.7	4±1	.03±.01	5±3	2±1	1±.1		4±.2	4±.2	3169	6422
	M2		1±.6	4±.4	3±1				4±.1	.6±.1			4474	3950
	M3		1±.6	1±.6	5±1				1±.1		5±.1	4±.2	7850	7065
A90	M1		1±.5	5±.3	3±1		.9±.4	1±1	.6±.1		1±.1	1±.1	3852	2413
	M2		1±.6						.7±.2				1983	1708
	M3		2±1	1±.3	5±2				2±.1				1959	1581
	M4		1±.6	1±.8	4±2				.6±.2				2003	1760
B86	M1		2±1	1±.3	2±1	.02±.01		2±1	.6±.1				1600	1452
	M2		1±.7	1±.7	5±1				1±.2		2±.2		9519	8023
	M3		2±.7	4±.5	3±1				1±.2		1±.1		1628	1706
	M4				3±1				4±.1		1±.1	1±.1	1655	1574
C89	M1		1±.5	1±.5	4±1		.8±.3	3±2	.7±.1	1±.1			6997	5652
	M2				3±1				.2±.1	1±.1			4509	4082
	M3		1±.5	1±.3	4±1				.8±.1		1±.1	1±.1	5485	4107
	M4				6±1				.2±.1		1±.1		3415	2807
D96	M1		1±.7	1±.7	8±2		1±.7	2±1	1±.2		8±.2	8±.2	5594	4831
	M2				9±2				1±.2		8±.2	8±.3	4361	3389
	M3		1±.7	1±.7	8±2				.1±.2		7±.2	7±.2	4131	3389
D99	M1				9±2		.5±.3	2±1	1±.2		7±.2	7±.2	5643	4317
	M2		1±.7	1±.8	2±1				.7±.1		2±.1		2802	2496
	M3				4±1				4±.1	.6±.1	2±.1		4508	4608
	M4				2±1				.1±.1		2±.1	2±.1	3820	3583
									.6±.1		2±1	2±1	4489	3716

All results ppm dry weight basis except Fe (mg/gm) and Sc results are relative counts only

TABLE 7b
Sediment Core Section Analyses

Location	Core Cut	Date	Se (136)	Se (264)	Cr (320)	Au (412)	Au (657)	Au (764)	Ag (937)	Co (1173)	Co (1332)	Fe (mg/gm) (1009)	Fe (mg/gm) (1291)	Sc (899)	Sc (1120)
F102	M1	22/2/73			3±1	.05±.03		5±3		1±1		2±1	2±1	4312	4000
	M2		1±7	2±1	3±1					1±2		2±2	3±2	3267	2820
	M3				5±1					1±2		3±2	3±2	2929	2200
	M4		1±8		2±1					1±2		2±2		2607	1988
G	M1				3±1					1±2	1±1	2±1	2±2	2680	2515
	M2		2±1	.5±.4	4±1	.09±.03	.3±2			1±1	1±1	2±1		2607	1938
	M3			1±4	3±1	.04±.02				.5±.1		2±1	2±1	3558	2453
	M4		1±4	.3±.3	2±1					1±1	1±1	1±1	1±1	2885	2303
H	M1			2±7	10±2				7±2	2±2	1±1	3±1	3±1	26,629	21,428
	M2		3±6	1±6	14±2					2±2	2±1	3±1	3±1	29,807	24,954
	M3		3±7	1±7	12±2		1±5	.6±1		2±2		3±1	3±1	23,408	17,300
	M4		3±9	1±1	16±2		1±6			1±2		5±2	4±2	22,313	17,678
J99 199	M1		1±5	1±5	3±1			2±1		.7±.1		1±1		1570	1480
	M2			1±8	3±1					.3±.1		1±1		1187	1168
	M3			1±6	3±1		1±3			.5±.2		1±1		1580	
	M4				2±1		1±5	1±7		.5±.1		1±1		2761	2663

All results ppm dry weight basis except FE (mg/gm) and Sc results are relative counts only

Table 8

Sample	Date	Replicate	Lab Number	Se		Cr	Au	Ag		Co		Fe (mg/gm)	
				(136)	(264)	(320)	(411)	(658)	(885)	(1173)	(1332)	(1090)	(1291)
A 96 M1 A*	21 May 73	1	268	1.13±.13	1.61±.15	5.77±.28	0.32±.01	2.66±.24	0.71±.29	2.10±.05	2.45±.04	2.52±.04	2.60±.03
A 96 M1 B		2	272	1.11±.12	1.45±.22	5.20±.34	0.31±.01	2.65±.15	1.75±.28	2.28±.05	2.41±.04	2.26±.03	2.34±.03
A 96 M1 C		3	276	1.33±.12	1.31±.16	5.24±.33	0.27±.01	2.62±.22		2.10±.04	2.23±.04	2.37±.03	2.40±.03
A 96 M2 A		1	280		1.43±.14	7.00±.43	0.34±.01	3.40±.22		2.41±.06	2.69±.04	2.89±.04	2.90±.03
A 96 M2 B		2	284		1.39±.15	5.28±.25	0.37±.01	3.89±.21	2.31±.26	2.60±.05	2.65±.04	2.84±.04	2.85±.03
A 96 M2 C		3	288	1.89±.18	2.09±.19	7.77±.49	0.50±.01	3.95±.22	1.45±.37	3.28±.06	3.38±.05	2.99±.03	3.10±.04
A 96 M3 A		1	292	2.21±.20	2.07±.35	8.02±.30	0.45±.02	4.06±.28	0.70±.35	3.15±.07	3.21±.05	3.30±.04	3.41±.04
A 96 M3 B		2	296	1.72±.13	2.02±.17	7.00±.44	0.45±.01	4.12±.28	1.72±.32	2.79±.06	2.91±.05	2.84±.03	2.89±.03
A 96 M3 C		3	300	3.29±1.6	1.98±.21	7.87±.42	0.38±.02	3.41±.23	0.53±.31	2.65±.05	2.90±.05	3.39±.03	3.31±.03
A 96 M4 A	21 May 73	1	304	1.86±.14	2.06±.25	6.97±.32	0.43±.01	3.79±.24	1.32±.36	2.84±.06	2.99±.04	3.24±.03	3.32±.09
A 96 M4 B		2	308	2.13±.16	1.36±.16	7.55±.38	0.36±.01	2.92±.19	0.96±.27	2.23±.04	2.45±.04	3.96±.04	3.81±.03
A 96 M4 C		3	312	1.69±.13	1.35±.18	6.07±.23	0.31±.02	2.91±.25	0.04±.31	2.23±.05	2.42±.04	4.34±.04	3.11±.03

*Each sample, e.g. A-location on Grid was divided into 3 aliquots

96-depth of water in ft.

A, B, C for analysis as

M₁, M₂, M₃, M₄-slice of core

separate samples.

Table 9 shows a comparison of results from radioactivation of ores and fertilizers with the results from AA and wet chemistry. The two different methods of analysis, A.A. and NAA, at two different laboratories give parallel results. Their ratio was $AA/INAA = 1.72 \pm .34$.

TABLE 9. CADMIUM CONTENT OF PHOSPHATE ORE AND FERTILIZER

Lab #	Sample	Vial	NAA ^a	AA
33802	fertilizer	1	24	42
38803	fertilizer	2	49; 48 ^b ; 47 ^b	86
38804	fertilizer	3	34	80
38807	fertilizer	4	50	108
38808	fertilizer	5	45	84
38812	fertilizer	6		50
38813	fertilizer	7	59; 68 ^b	114
38815	fertilizer	8	111	154
38820	fertilizer	9	16	20
38821	fertilizer	10	102	140
38822	Ore	11	76	106
38826	Ore	12	90	161

^a results are $\mu\text{g Cd}$ per gram sample, dry weight.

^b same standard but counted at a later time.

SECTION VII

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